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THE LUBRICATING CHARACTER OF GREASES USING A NEW TEST METHOD.(U)  
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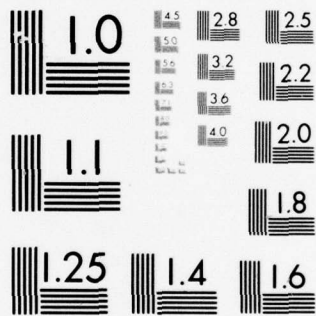
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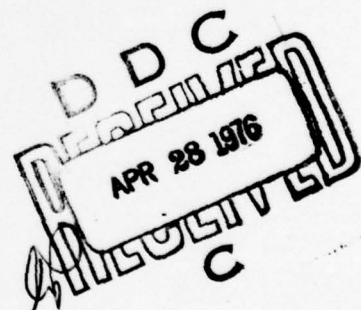
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4 April 1977

A FINAL REPORT ON THE LUBRICATING  
CHARACTER OF GREASES USING A NEW  
TEST METHOD

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By

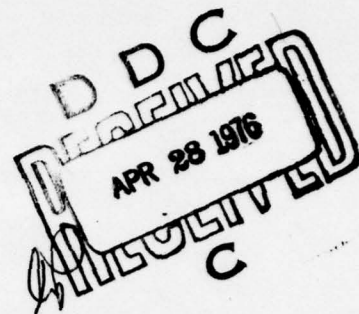
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Using a new slow-speed high frequency reciprocating test rig, tests have been made on a series of PTFE and ammeline greases.



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20. Abstract A slow-speed high frequency reciprocating test rig was used to investigate the behavior of a lubricated sliding contact when subjected to repeated slow-speed passes within times comparable to the reaction rates of chemically acting lubricant additives. Such conditions are similar to the working environment of a lubricant and tests carried out using this test rig are shown to reveal more information than may be obtained otherwise. The report presents a brief description of the test rig and the results of tests made on a series of ammeline and teflon greases using this new method. The results of tests on a commercial mineral oil are also included		

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### SUMMARY

At Imperial College we have designed and developed a laboratory test rig to investigate the behaviour of a lubricated sliding contact when subjected to repeated slow-speed passes within times comparable to the reaction rates of chemically acting lubricant additives. Such conditions are often akin to the working environment of a lubricant and tests carried out on this rig are shown to reveal more information than may be obtained otherwise. This report presents a brief description of this rig and the results of tests made on a series of ammeline and PTFE greases using this new method. The results of tests on a commercial mineral oil are also included.

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## Introduction

In 1972, an investigation was carried out at Imperial College, London, using various laboratory rigs to test the effects of extreme pressure and detergent additives on the load carrying and wear resistance characteristics of a mineral oil base stock. The results showed that an e.p. additive has little effect on the scuffing resistance of the lubricant as measured in both a reciprocating piston-ring scuffing rig and a working internal combustion engine. This was considered to be very significant since such behaviour was not seen in the normal laboratory tests developed for the evaluation of gear lubricants.

It was suggested that the high temperatures and pressures which prevail at T.D.C., and e.p. additive will react chemically with the metal surface of the liner to produce a protective boundary lubricant. In a reciprocating piston engine, however, that area of liner immediately below T.D.C. is subjected to a second wipe by the piston-ring withing about 3 ms. This short time is comparable with the reaction rates of e.p. additives. Assuming collapse of the squeeze-film and that the entire boundary layer might be removed by the first upward wipe, there may be insufficient time for its replacement before the second wipe and so a scuff would ensue.

Such considerations were also thought to be applicable to the failure of high speed gears and other metal-pairs subjected to repeated contact at high frequencies. It was therefore suggested that the theories developed should be decked by some new type of laboratory test rig, the important design criteria of which being that firstly sliding speeds should be slow enough to prevent E.H.L. and the generation of high contact temperatures and secondly that

frequencies up to 150 Hz and temperatures up to 350°C. should be attainable.

The following sections of this report include a brief description of a rig which fulfills these criteria, the results of some preliminary tests on a commercial engine oil, and, finally, an investigation, using this new test method, of the lubricating character of a series of PTFE and ammeline greases supplied by the USAF.



### General Description of the Apparatus

A metal pin is loaded vertically and vibrated horizontally against a metal flat. The pin is held in a chuck driven by a vibrator and is loaded against the flat by a weight hanging freely below the point of contact. The flat rests in an oil-bath which is supported to one side by a force-guage and from above by four stringers bolted to the frame of the rig. A small but powerful oven encloses the whole.

Friction is measured using the force-guage and electrical contact resistance by the potential drop of a 15mv voltage source applied across the contact. A simple displacement transducer monitors chuck movement.

### The Chuck and Driving-Rod

Machined from stainless steel, the chuck is a simple jaw vice closed by a single countersunk screw. Its sides project far enough to allow the loading wires to run either side of the chuck. The driving-rod, also of stainless steel, is  $\frac{1}{4}$  in. diam., 6" long, and is screwed by one end to the chuck, by the other to the core of the vibrator. Sandwiched between the rod and the chuck, a thin guage stainless steel plate holds the core of the displacement transducer.

### The Oil Bath

$1\frac{1}{2}$  in. square and  $\frac{1}{4}$  in. deep, the oil bath together with its connecting flange was milled and turned from a

solid stainless steel block to ensure great strength combined with low mass. The specimen rests between two steps cut inside and is held secure by two set-screws. The four ends of two V-shaped stringers, whose apexes are screwed to opposite ends of the bath, are bolted to the rigid overhead team of the frame and prevent any movement of the bath other than in the direction of the friction force.

#### The Oven

The oven, made up of 5 pieces of cyndanio sheet, is powered by two 450w. strip-heaters. The bottom is left open to allow the loading wires to run freely to the weight hanging below and holes cut in the walls accommodate the driving rod and transducers.

#### The Frame

Welded 2 in. x  $1\frac{1}{2}$  in. box-section steel is used throughout to ensure rigidity and absence of unwanted resonance. The entrie structure stands 3 ft. high so allowing the loading weight to hang a suitably large distance below the contact. The oil-bath and oven hang from a 3 ft high overhead beam. To one side another beam,  $2\frac{1}{2}$  ft high, holds the bodies of the two transducers and to the other side, a platform for the vibrator.

#### The Vibrator

Powered by a Ling oscillator/amplifier the Goodmans 100 watt vibrator can drive the chuck with a stroke of

1 mm up to a frequency of 200 Hz.

### The Force-Guage

The body of the force-guage is bolted to the frame and the core is heat-insulated from the oil bath by a cyndanio sheet at their connecting flange. A piezo-electric transducer, it measures force direct with negligible displacement so inertia effects and phase-lag due to the mass of the system are insignificant.

Resolution is high, but for low impedance output loads a charge amplifier must be used.

### The Displacement Transducer

A simple linear inductive device, whose core is screwed to the chuck, monitors the motion of the driven specimen.

### 15 mV Voltage Source

A 15 v., 100 MA power supply is connected to a 10:1 potential divider of three different impedences, the small resistors being 1, 10 and 100 ohms respectively. The 15 mV. voltage across any one of these resistors is applied across the contact and by matching their impedences a rough measurement of the contact resistance is made.

### The Specimens

The chuck and oil-bath are designed to accept both 'pin on flat' specimens (of any metallurgy, of course) as



well as 'piston-ring on liner' specimens.

#### Recording Equipment

The output of the force-guage, measuring force, is basically a square-wave with a superimposed high frequency component, whilst the contact resistance is evident as a steady voltage with sharp drops to zero at asperity contacts. The temperature is measured using a thermocouple placed closed to the point of contact.

A number of different recording techniques have been used, namely (a) direct measurement of friction and contact resistance from an oscilloscope screen and temperature from a microvoltmeter, (b) string these three parameters on magnetic tape using a two-channel tape-recorder (the friction and contact resistance being frequency modulated before recording alternately on one channel and the temperature being 'spoken over' on the second channel) (c) using a pen-recorder (the friction force being first fully-rectified and smoothed) and (d) a U.V. recorder.

## Materials

### (1) Simulated Crankshaft Bearing

The test pegs were manufactured from EN16T manganese-molybdenum, crankshaft steel, for which there is no equivalent SAE and Al21 number. The specified composition (BS 3179) is shown in the table.

C (%)	Si (%)	Mn (%)	Mo (%)	S (%)	P (%)	Fe (%)
0.3-0.4	0.1-0.35	1.3-1.8	0.2-0.35	0.050 max	0.050 max	remain- der

The bearing material consisted of a strip of VP 10 leaded bronze alloy without overlay, supplied by Vandervell Products Ltd. The composition is shown below.

Pb (%)	Sn (%)	Fe (%)	Cu (%)
10	10	0.5	remainder

## Tests

Using this rig tests have been carried out on (a) a commercial mineral oil and (b) a series of PTFE and ammeline thickened greases supplied by USAF.

### (a) Mineral Oil

In this laboratory, tests have been made on a commercial mineral oil using a Bowden-Leben machine ( ). This measures the friction force between a very slow moving peg and a flat immersed in the oil under test. The peg makes a single slow speed pass at 0.05 mm/s whilst the oil is slowly heated from room temperature to 300°C.

The test pegs were manufactured from EN16T manganese-molybdenum crankshaft steel. The flat consisted of a strip of VP10 leaded bronze alloy without overlay and was supplied by Vandervell Products Ltd.

Keeping the metallurgy and pressure of the contact the same, this oil was then tested using the new rig. The pin was moved at a frequency of 20 Hz or above and a stroke of 3/100 in. Otherwise the test procedure was no different from that of the Bowden-Leben tests. The results showed repeatable plots of friction vs. temperature that were markedly different from those of the Bowden-Leben tests.

## Results

Figs (1) and (2) show pen recorder traces from both tests respectively and Fig (3) the two friction vs. temperature plots together. Whilst up to 212°C, the Bowden-Leben tests showed a steady fall in friction-coefficient the HFR (High-frequency reciprocating) rig tests showed a transition at 55°C with subsequent recovery at 70°C, a steady increase at 100°C and a further transition at 150°C. Above 225°C both showed similar friction increases. At 260°C however, only the vibration rig showed a recovery.



## Discussion

Considering the Bowden-Leben tests, we can assume that the transitions in friction above 212°C are due to e.p. additive action, but below this temperature we see only a steady decrease in friction from which little if any information can be divulged. The HFR tests however show a number of transitions below 212°C and from these we can infer considerably more information about the additive behaviour of the oil.

The transition at 55°C is caused by the peg repeatedly wiping off a layer of alkylamine corrosion inhibitor which at this temperature starts 'melting'. The recovery at 70°C marks that temperature at which the surface is sufficiently clean to allow the physical adsorption of surfactant molecules. These are in dynamic equilibrium with the bulk of the oil and so the surface coverage decreases with temperature. This might explain the gradual increase in friction above 100°C. At 150°C the surface coverage becomes insufficient to sustain a protective boundary layer and a transition occurs. This is only apparent in the HFR test results because the successive passes of the peg allow insufficient time for any chemical reaction with the specimens. The subsequent friction changes can be explained by the action of e.p. additives whose reaction rates at these temperatures are now fast enough to maintain a protective boundary layer over most of the stroke. The recovery at 260°C is only apparent on the HFR rig plot because the peg wipes the surface clean and so allows further reactions to occur.

### (b) Greases

The USAF have supplied the laboratory with nine PTFE thickened greases, nine ammeline thickened greases and the perfluoralkyl ether base oil common to both. A programme of tests using a number of different methods have been carried out to determine the lubricating

character of these greases.

### Results

Fig (4) shows the pen recorder trace from a test on the base oil. The results of tests on the greases however, were stored on tape and played back later through an oscilloscope. Figs (5) and (6) show friction vs. temperature plots for the PTFE and ammeline thickened greases respectively. The friction coefficient of the base oil fell steadily with increasing temperature until  $240^{\circ}\text{C}$  when a transition occurred. Fig (5) shows that the overall friction of the PTFE greases was lower than that of the base oil, especially at low temperatures, and that it took about five minutes at room temperature for the friction to <sup>reach</sup> a steady value. Unlike the base oil, the greases exhibited various friction changes between  $125^{\circ}\text{C}$  and  $175^{\circ}\text{C}$  and their final transitions in friction all occurred at higher temperatures.

Fig (6) shows that the ammeline thickened greases also underwent friction changes between  $125^{\circ}\text{C}$  and  $175^{\circ}\text{C}$ . However, they showed no increase in scuff resistance at high temperatures.

None of the greases exhibited any dependence on the number of times they were passed through the homogeniser.

### Discussion

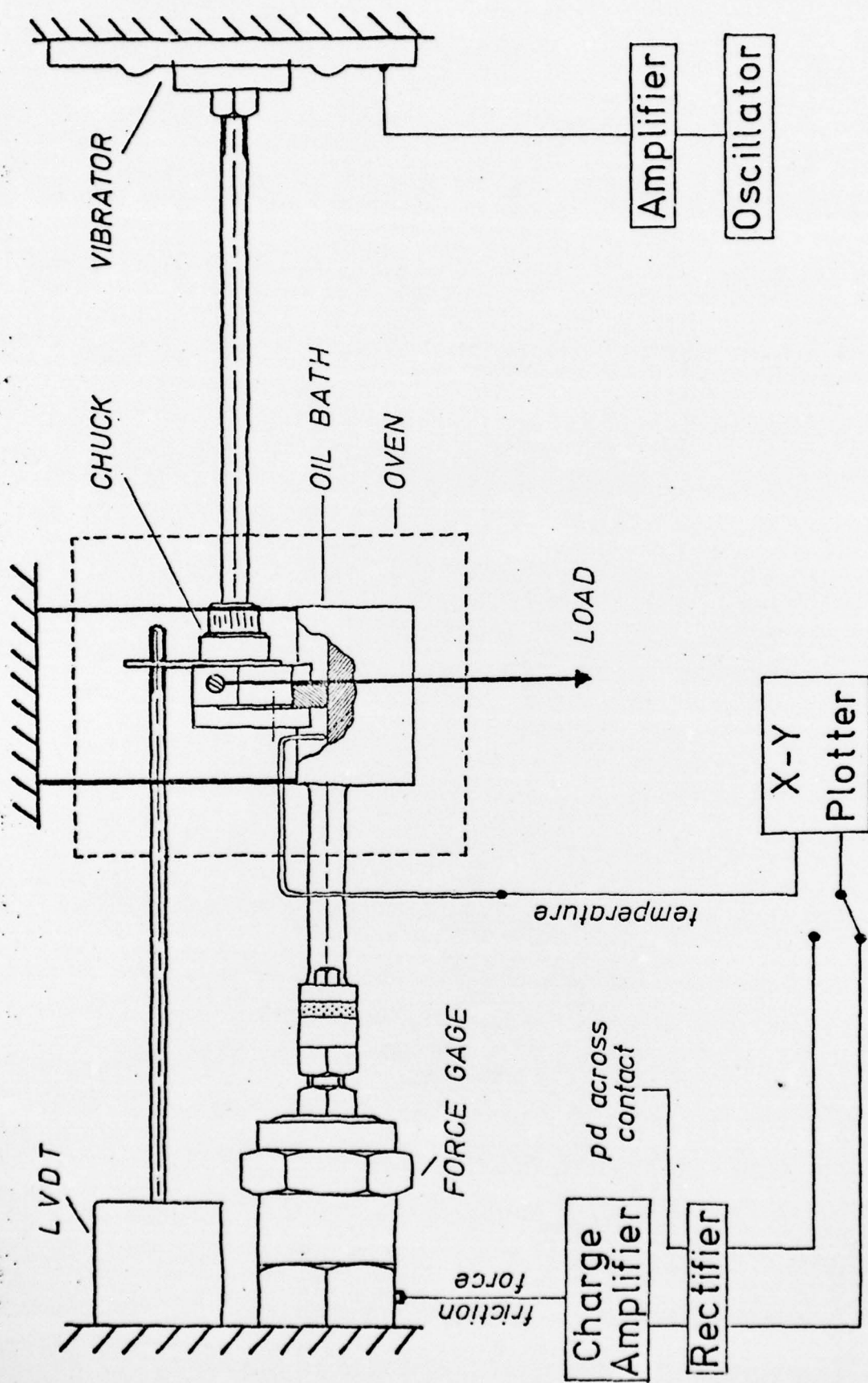
The optical section of this study (1) showed that the film thickness increased as the grease was worked in the contact. This was matched by the decrease in friction coefficient during the first five minutes running in the reciprocating rig. Unlike the ball on glass disc tests, however, this effect was not repeated when restarting after a few minutes pause. This behaviour might be explained by a thickening of the grease with working and/or a layer of thickener particles building up on the surfaces.

The low temperature friction coefficient of the greases is markedly lower than that of the base oil due to a physical reaction between the PTFE or ammeline and the metal substrate. This causes a thin layer of low shear strength to adhere to the rubbing surfaces. At about 150°C, however, the molecular vibrations of this layer cause it to desorb resulting in a change of friction. The subsequent recoveries must be caused by some e.p. action e.g. free sulphur present in the base oil.

Figs (5) and (6) show that the base oil scuffs at 250°C and only the PTFE greases raise this temperature. (20% PTFE by as much as 50°C.)

Previous work (2) has shown that at high temperatures the PTFE chain, is broken. Consequently the free radicals formed produce various reaction products. Chemical reactions, initiated by the free radicals and partially catalysed by the clean metal surfaces which are developed during running, produce a polymer. This polymer has the property of modifying the rubbing surfaces and this together with other reaction products acts as a scuff inhibitor. It is however, highly corrosive, and so increasing PTFE concentration tends to diminish its beneficial effect.





THE HFR RIG

FIG 1 MINERAL OIL,  
BOWDEN-LEGEN

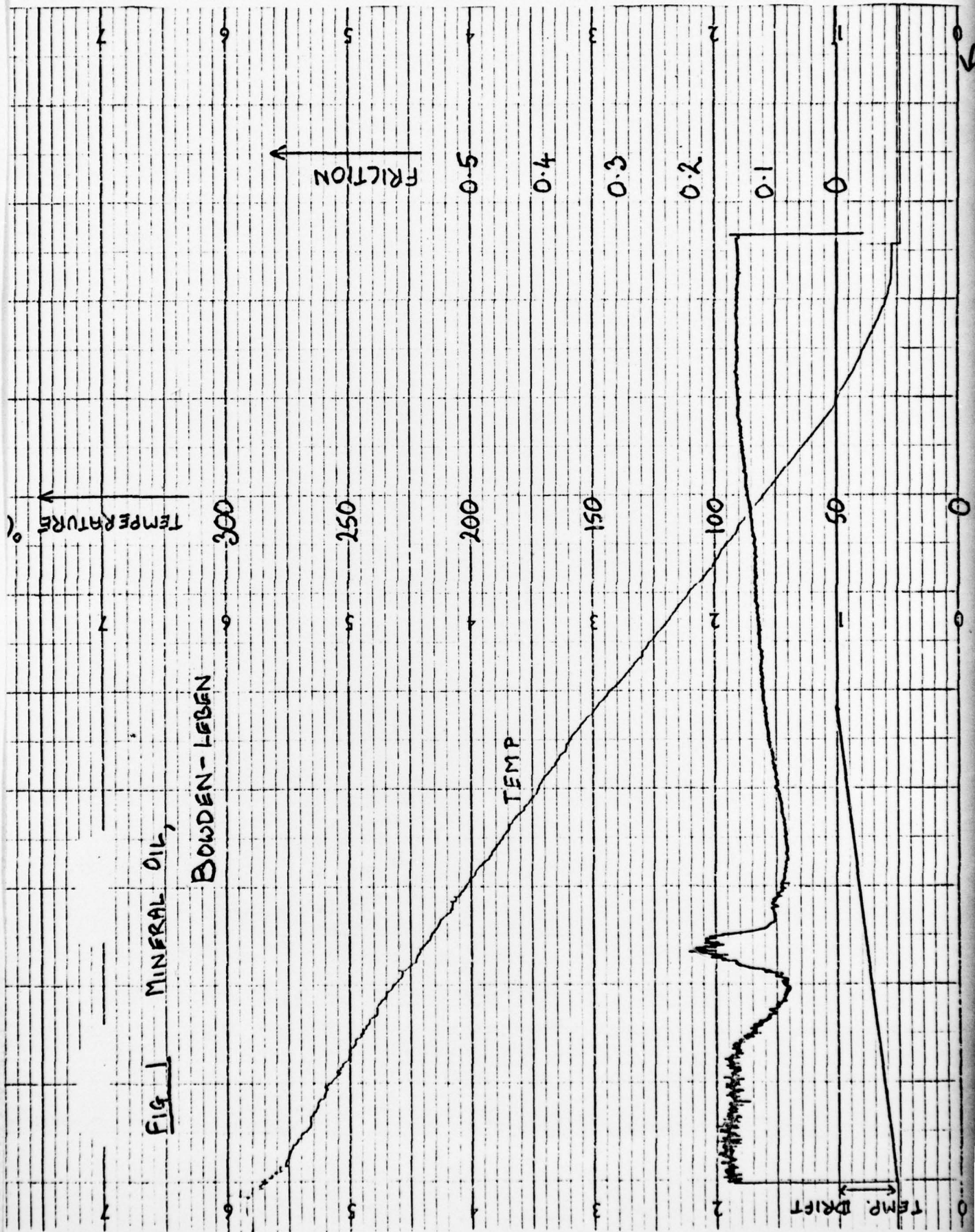


FIG 2 MINERAL OIL, VIBRATION TEST

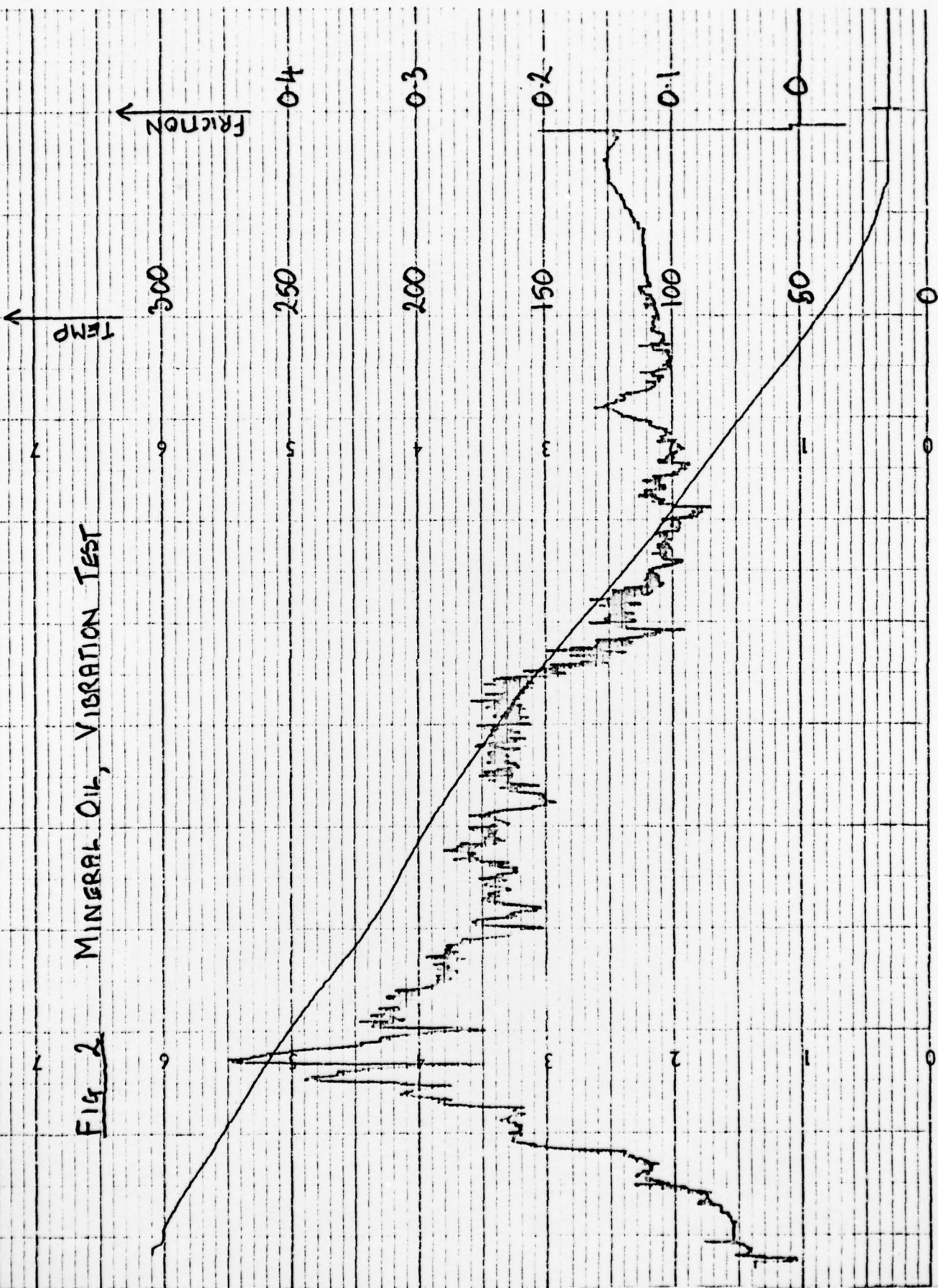




FIGURE 3 MINERAL OIL

$\mu = 0.45$

○ BOLDEN-LEBEN TESTS

□ VIBRATION TESTS

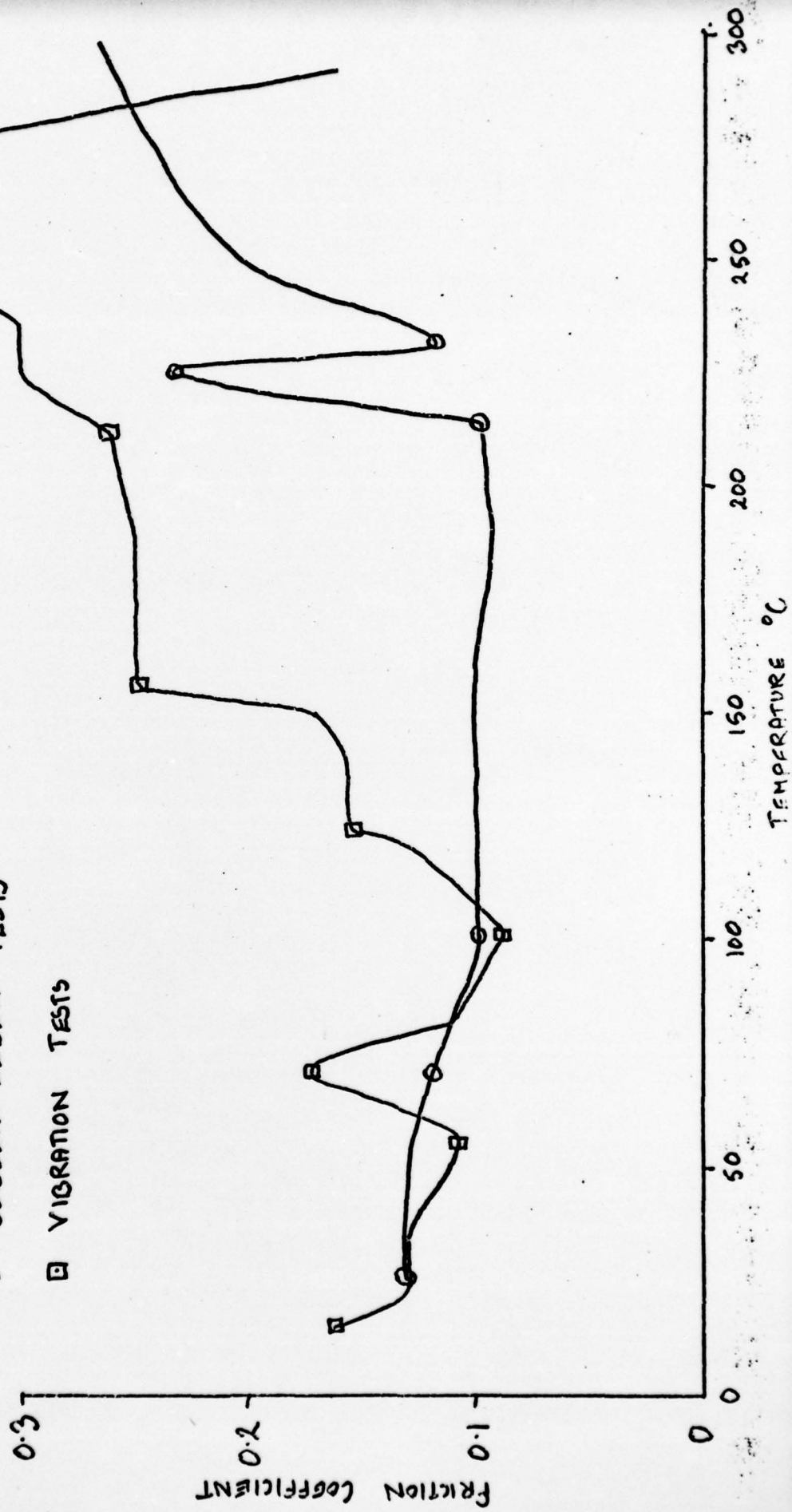


FIG 4

BASE OIL,  
VIBRATION TEST

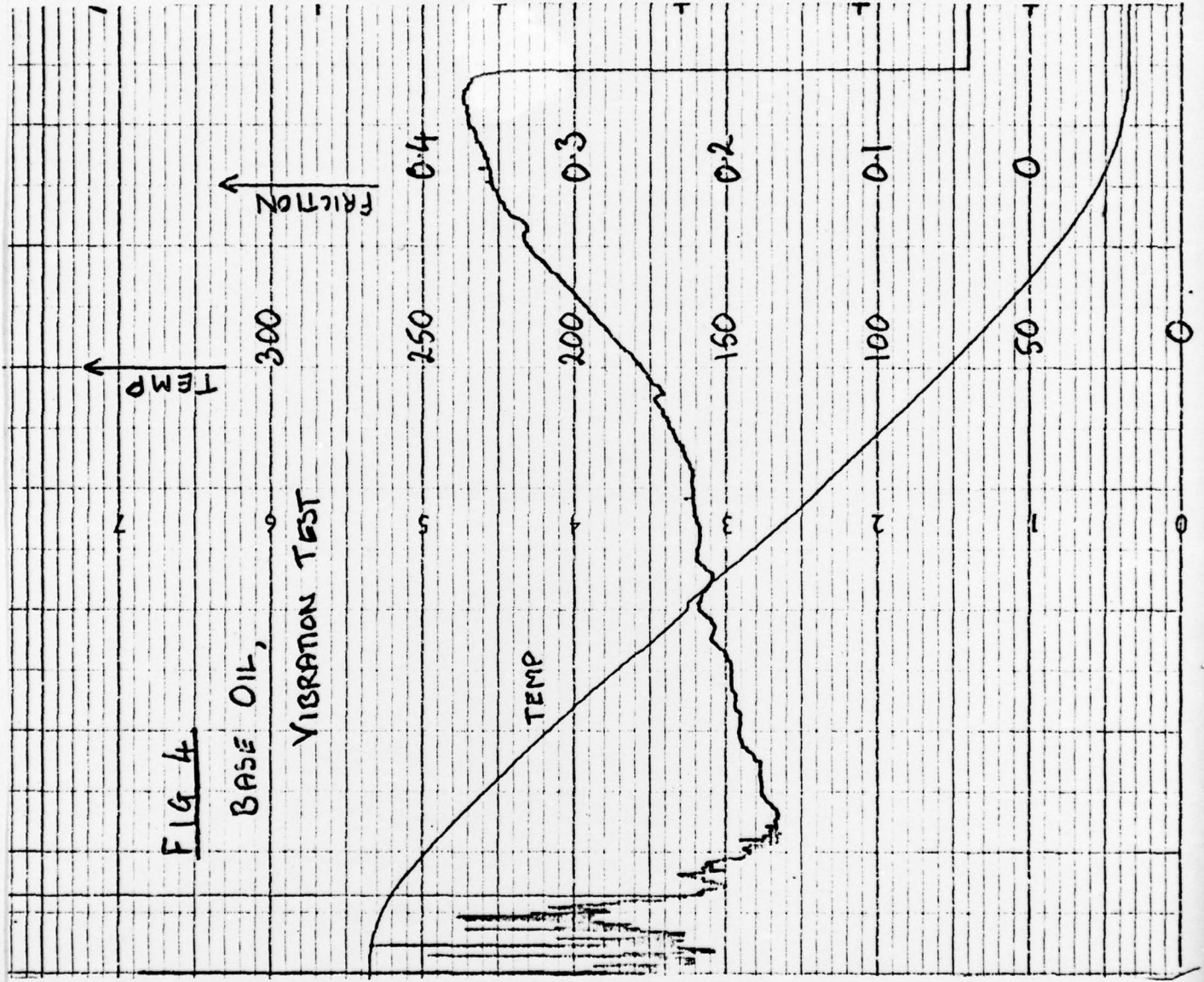


FIGURE 5 PTFE GREASES, VIBRATION TESTS

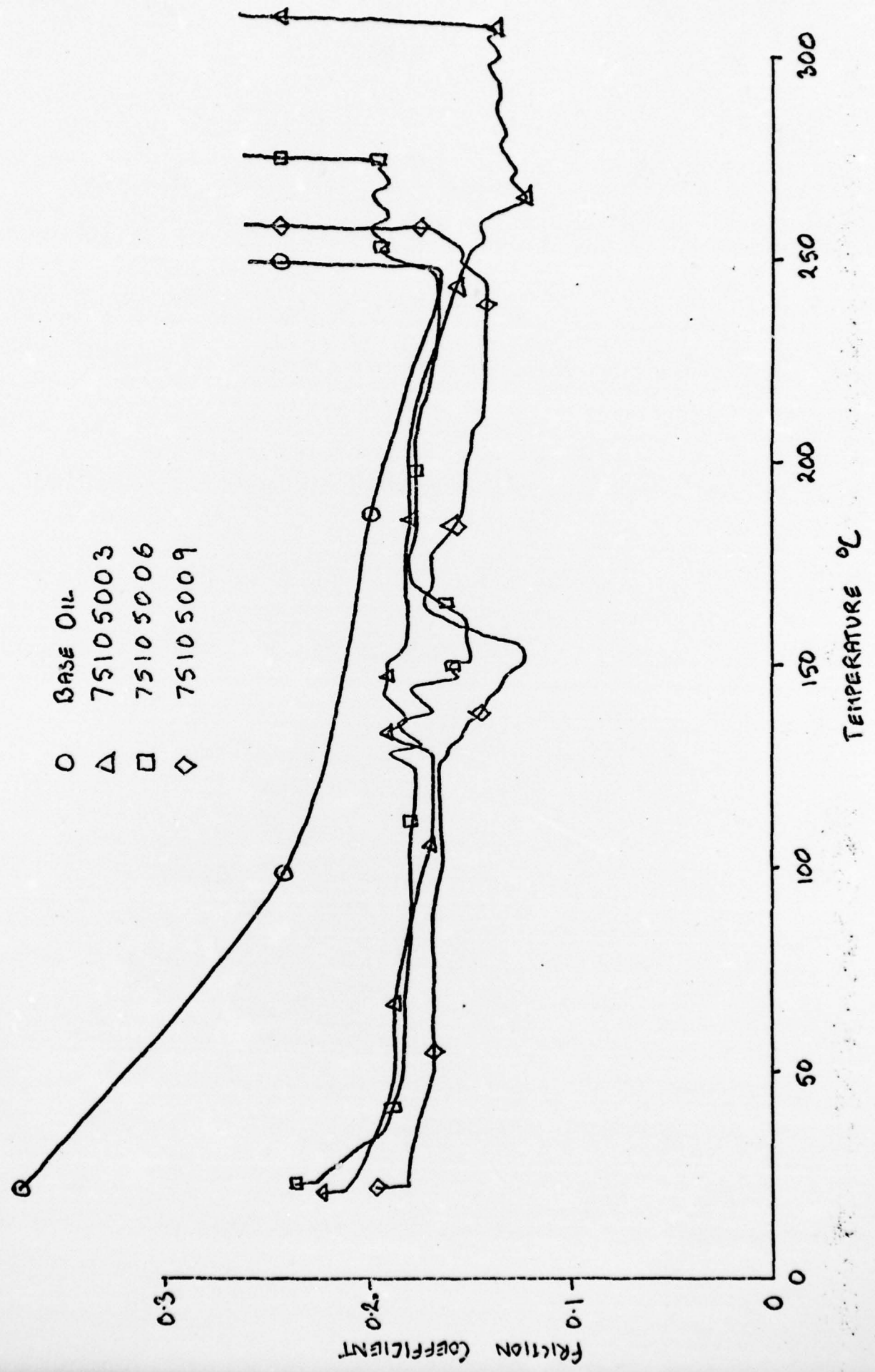
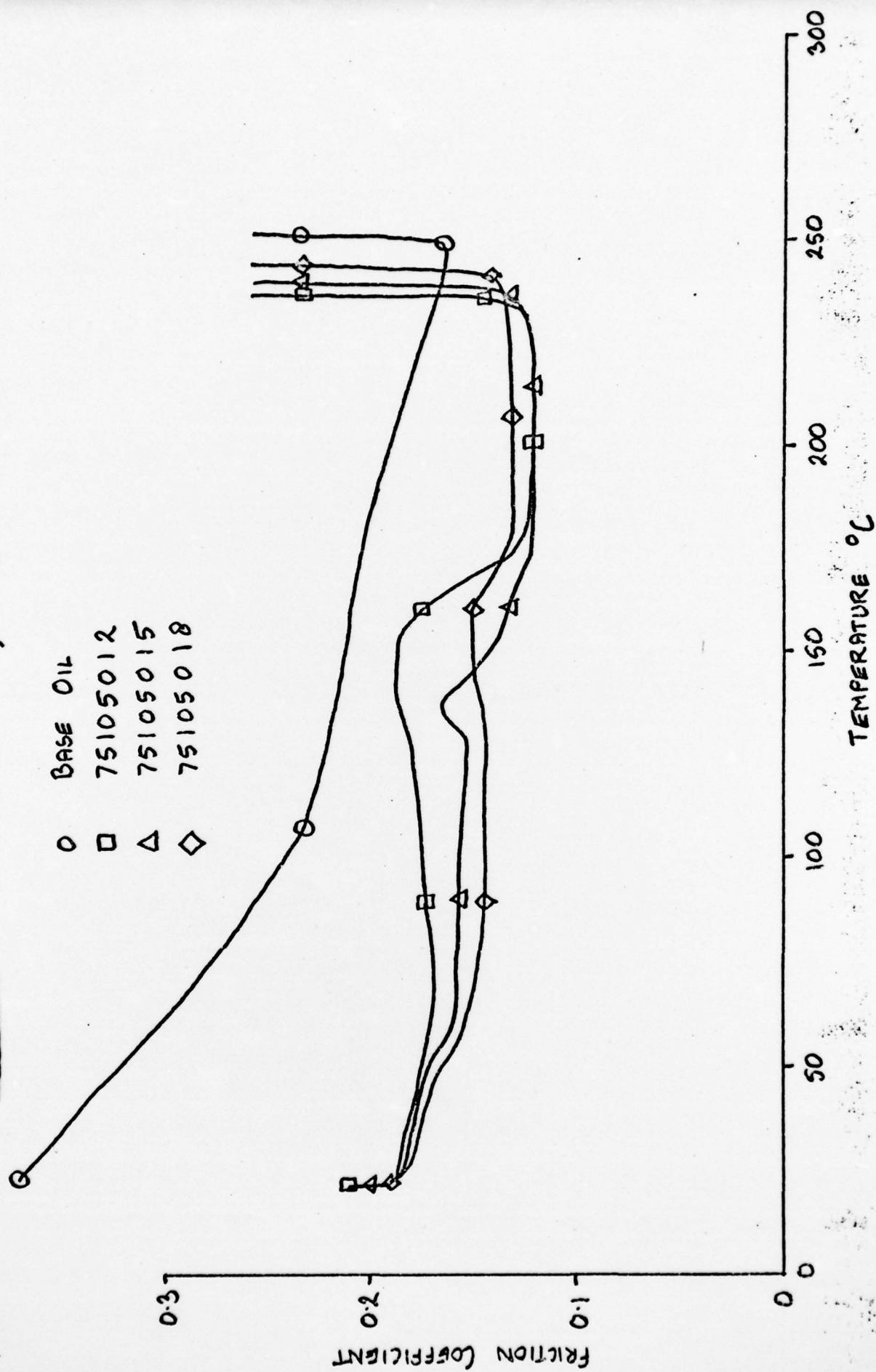


FIGURE 6 AMMELINE GREASES, VIBRATION TESTS

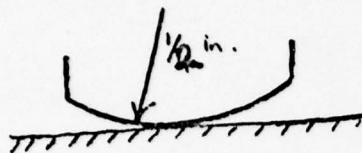




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1. The Lubricating Character of Greases, Optical Section.  
Lubrication Lab., Imperial College, 7 April 1976.
2. Remarks on the wear of a journal bearing lubricated by a grease  
containing a powdered PTFE additive.  
T. Stolarski. Tribology Int. August 1976.

# Appendix I : Maximum Flash Temperature



$$\text{Frequency} = 50 \text{ Hz}$$

$$\text{Load} = 2 \text{ lb.}$$

$$\text{Stroke} = 30 \text{ thou.}$$

$$\text{Friction Coefficient } \mu = 0.1$$

$$\begin{aligned} \text{Radius of contact, } a &= 3.43 \times 10^{-3} (RW)^{1/3} \text{ for steel} \\ &= 3.43 \times 10^{-3} \left(\frac{1}{2} \times 2\right)^{1/3} \\ &= 3.43 \text{ thou.} \end{aligned}$$

$$\begin{aligned} \therefore \text{Width of contact} &= 2 \times 3.43 \\ &= 6.86 \text{ thou.} = 0.0174 \text{ cm.} \end{aligned}$$

$$\begin{aligned} \text{Maximum speed, } u_{\max} &= \pi \dot{\theta} \\ &= \frac{30 \times 10^{-3}}{2} \times 2.54 \times 50 \times 2\pi \\ &= 12 \text{ cm/s.} \end{aligned}$$

$$\begin{aligned} \text{Heat generation, } q &= \frac{\mu W u}{\pi a^2} \\ &= \frac{0.1 \times 2 \times 9.81 \times 0.12}{\pi (3.43 \times 10^{-3} \times 2.54)^2} \text{ watts/cm}^2 \\ &= 105 \text{ cal/s/cm}^2 \end{aligned}$$

$$\begin{aligned} \text{Maximum temperature, } T_{\max} &= \frac{q}{K} \left( \frac{Lk}{\pi u} \right)^{1/2} \\ &= \frac{105}{0.11} \left( \frac{0.0174 \times 0.12}{\pi \times 12} \right)^{1/2} \\ &= \underline{\underline{6^\circ \text{C}}} \end{aligned}$$



# APPENDIX II

MCG No.	THICKENER CONCENTRATION	NUMBER PASSES THROUGH HOMOGENIZER	PENETRATIONS			PARTICLE DISTRIBUTION	
			0 STROKE	60 STROKES	10,000 STROKES	RANGE MICRONS	AVERAGE SIZE, MICRONS
75105003	20% PTFE	5	335	369	350	0.375 - 1.315	0.750
75105004	20% PTFE	10	337	332	341	0.440 - 1.440	0.815
75105005	20% PTFE	15	335	337	350	0.500 - 1.375	0.875
75105006	25% PTFE	5	298	333	343	0.500 - 1.190	0.690
75105007	25% PTFE	10	305	309	320	0.375 - 1.065	0.650
75105008	25% PTFE	15	305	318	332	0.375 - 1.1563	0.685
75105009	30% PTFE	5	268	286	298	0.565 - 1.030	0.720
75105010	30% PTFE	10	264	290	309	0.440 - 1.125	0.690
75105011	30% PTFE	15	262	283	298	0.440 - 0.995	0.700
75105012	15% Ammeline	5	345	352	+410	0.565 - 5.125	1.210
75105013	15% Ammeline	10	309	332	388	0.4375 - 2.940	1.075
75105014	15% Ammeline	15	296	318	343	0.500 - 3.065	1.075
75105015	20% Ammeline	5	347	330	344	0.690 - 4.565	1.275
75105016	20% Ammeline	10	311	328	324	0.565 - 5.375	1.325
75105017	20% Ammeline	15	297	315	318	0.565 - 4.250	1.205
75105018	25% Ammeline	5	337	343	333	0.625 - 4.250	1.400
75105019	25% Ammeline	10	296	299	296	0.500 - 4.3125	1.385
75105020	25% Ammeline	15	282	294	287	0.500 - 2.000	1.055